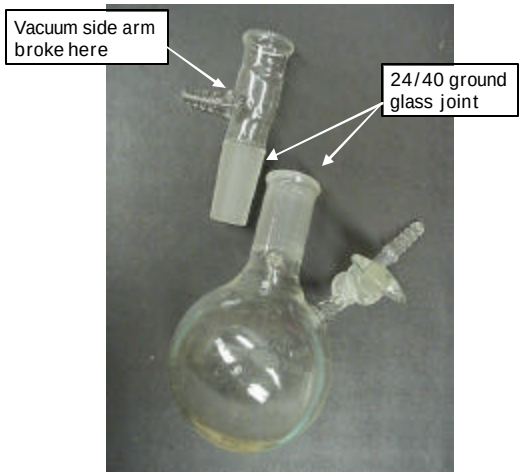
LBNL Lessons Learned Statement
Frozen Glassware Joint Leads to Breakage and Injury

1 August, 2001

Background:

An employee synthesized an organic compound that was dissolved in methyl alcohol (methanol). At the end of her procedure, she placed the solution into a 250-ml round bottom boiling flask (part of a Schlenk apparatus) and, through several steps, gradually removed the methyl alcohol. This required that a vacuum adapter be placed in the neck of the boiling flask. The two mated using a standard taper 24/40 ground glass connection, which she had lubricated using stopcock grease (standard procedure).



Typical Schlenk Flask w/ Vacuum Adapter

At the end of the evaporation she removed the flask/adapter from the vacuum apparatus and attempted to separate the ground glass joint. It was frozen (would not separate). In an attempt to separate the fitting, she twisted the connection and the side arm on the vacuum adapter broke, causing a puncture and laceration to her right hand. This wound later became infected and resulted in the employee being hospitalized for a short time for intensive antibiotic treatment.

Analysis:

Glassware lacerations and punctures are among the most common accidents in chemistry laboratories. There are several issues with respect to this particular accident that should be noted:

1. Although the employee lubricated the ground glass joint with stopcock grease, she did not recognize that many organic solvents, especially methyl alcohol and

hexane, can dissolve the grease. What apparently contributed to the joint getting frozen was that the methyl alcohol vapor in the flask dissolved some of the grease that had been placed in the joint.

2. There are a number of ways to free frozen joints that are preferable to twisting the fittings, and one of these methods (see below) should have been used to disassemble the fitting.

3. Although the employee was wearing disposable laboratory gloves, these did not provide her with physical protection against the broken glass.

Corrective Actions/Suggestions

1. Use sufficient stopcock grease to assure that the joint will separate. When organic solvents are used in the glassware, particularly when they are refluxed, consider applying additional grease on the male portion of the fitting.

2. If a fitting will not separate easily, ***DO NOT FORCE IT BY TWISTING HARDER.*** Consider discarding the material and the fitting; this may be preferable to running the risk of breaking the glassware and causing injury.

   If the fitting must be separated, use one of several alternative methods:

   - Rock the fitting gently from side to side rather than twisting it. It may be helpful to place a bit of solvent around the top of the joint to try to work it down into the joint as the joint is rocked. *Note: wear heavy leather gloves when this is attempted.*
   - Apply gentle heat, carefully increasing if necessary. Hot water or steam jet is preferable to open flame, but if open flame is used (absolutely the last resort) it should be a cool (yellow) flame, and the joint must be rapidly rotated in the flame. *Note: do not use heat on any closed system, and consider the properties of the contents of the system when deciding whether to use heat.*
   - Soak the joint overnight in a basic solution (alcoholic KOH cleaning solution, for example). Note: some researchers have reported that soaking overnight in Coca Cola® (carbonated, weakly acidic solution) has succeeded in loosening a frozen joint. Use of an ultrasonic cleaner may also help.
   - As a last resort, a "Bottle Stopper Remover" (catalogue number 02927 as of 7/20/2001) is available from Fisher Scientific.

3. Whether heat is applied or not, wear heavy leather gloves whenever unusual force is required to separate a ground glass joint. If a fitting does break during the separation the gloves will protect the hands.

4. Report all injuries, no matter how minor, to LBNL Health Services, Building 26, x6266.

Further Information

"The Glassware Gallery" (http://www.ilpi.com/inorganic/glassware/ as of 1 August 2001) has information about chemistry glassware, as well as an excellent discussion about stuck joints.

For further information on this incident please contact John Seabury, LBNL Environment, Health and Safety, at extension 6547.